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UNITED STATES DEPARTMENT OF AGRICULTURE

Double Superphosphate

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CONTENTS

	Page
Importance of double superphosphate	1
Definition of double superphosphate	2
Processses	3
Patents	4
History of production abroad	5
History of American production	6
Production statistics	9
Imports	10
Exports	11
Uses	11
Consumption	13
Agricultural value	16
Chemical composition	17
Physical properties	21
Literature cited	22

IMPORTANCE OF DOUBLE SUPERPHOSPHATE

Although double superphosphate has been manufactured since 1872, little has been written about it and it was almost unknown to farmers until recent years. Improvements in processes for making double superphosphate and an increasing realization of its advantages have greatly increased its use in recent years. Although the production of this fertilizer in the United States had never exceeded 100,000 tons in any year until 1936, it had increased to 329,046 tons in 1940 and since then has remained at an annual rate of more than 300,000 tons. Despite this increase the demand has exceeded the supply in the past 2 years. In 1942, a total of 143,000 tons was used in raising the grade of ordinary superphosphate and in mixed fertilizers, 156,000 tons was commercially distributed for direct use in agriculture, 21,000 tons was used in experiments and demonstrations in cooperation with the Tennessee Valley Authority, and 10,000 tons was distributed by the Agricultural Adjustment Administration. A very large tonnage was also exported.

Recent experiments with a variety of crops grown on many different types of soil in a large number of States show that equivalent quantities of available phosphoric acid (P_2O_5) in the form of double superphosphate give approximately the same increases in yield as are obtainable with ordinary superphosphate. The average grade of double superphosphate in 1942 contained 46.5 percent available P_2O_5, or more than double the 19 percent found in the average grade of ordinary superphosphate. Thus, transportation, bagging, and labor charges are relatively lower on phosphoric acid in the form of double superphosphate than in other forms; consequently the unit cost of phosphoric acid to the farmer in some regions of the United States is lower in the superphosphate form than in any other readily available fertilizer. Double superphosphate has decided advantages over other phosphoric materials (1) for making certain high-analysis mixed fertilizers; (2) for direct use in agriculture in the United States at points distant from a superphosphate plant; (3) in the utilization of certain grades of phosphate rock unsuitable for manufacture of ordinary superphosphate; and (4) in the export market. It is also the ideal material for raising the grade of run-of-pile ordinary superphosphate.

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[1] Credit is due K. D. Jacob, of this Division, for valuable suggestions and criticisms in the preparation of this report.

For a long time statistics relating to the manufacture and consumption of double superphosphate and more information on its properties and value in agriculture have been needed. The war has greatly increased this need. In recent years the activities of the Agricultural Adjustment Agency, the Tennessee Valley Authority, the War Food Administration, and the office of Lend-Lease Administration have all involved problems related to the manufacture and use of double superphosphate. It is important to use our resources to best advantage at any time; in time of war, it is doubly important.

The history of the development of the manufacture and use of double superphosphate has never been written, insofar as can be learned. Nearly all persons connected with its early development are now dead. It seems desirable, therefore, to ascertain and record the principal facts while it is still possible to do so. Many of the facts and figures recorded in this circular are published for the first time.

DEFINITION OF DOUBLE SUPERPHOSPHATE

The name "superphosphate" is a general term applied to various fertilizer products obtained by treating phosphate rock, apatite, bone, or other phosphatic materials with either sulfuric or phosphoric acid, or with a mixture of both. In order to distinguish superphosphate made by acidulation of phosphates with phosphoric acid from other types of superphosphate the specific name "double superphosphate" has long been used. In more recent years this material has also been called triple, treble, multiple, and concentrated superphosphate, Electrophos, and other names. The term "double superphosphate" is preferred to other designations, because (1) it is a literal translation of the original name, "doppel superphosphat," which referred to double acidulation of phosphate rock; (2) it is the name now used in other countries or a literal translation of the name there used for the same material, and thus its meaning is universally understood; (3) it was never called anything but double superphosphate in this or any other country, as far as is known, until half a century after it came into commercial use; (4) some of the others are trade names that are properly used only to specify a certain manufacturer's product; (5) double superphosphate usually contains 40 to 48 percent of available P_2O_5, which is about double the 18 to 20 percent of available P_2O_5 usually guaranteed in present-day ordinary superphosphate.

The names "triple" and "treble" had significance when first applied to double superphosphate because the usual grades of ordinary superphosphate then ranged from 12 to 18 percent. Although the terms "concentrated superphosphate" and "double superphosphate" are sometimes used as if synonymous, the first has a broader significance than the second. The name "concentrated superphosphate" has long been used to designate any phosphatic material containing more than 25 percent of available P_2O_5, whereas the name "double superphosphate" is properly applied only to the fertilizer material prepared by treating ground phosphate rock with phosphoric acid.

PROCESSES

Phosphoric acid is prepared commercially for use in making double superphosphate by both the wet and the furnace process. In the wet process finely ground phosphate rock is digested with dilute sulfuric acid and the resulting dilute phosphoric acid is decanted or filtered from the gypsum and concentrated by evaporation. In the furnace process an intimate mixture of phosphate rock, silica, and coke is heated to about 1,500° C., either in an electric or a blast furnace. Under these conditions most of the phosphorus is volatilized and is recovered either as elemental phosphorus by condensation or as concentrated phosphoric acid by oxidation and combination with water in precipitators, such as those shown in figure 1. In either case the second step in making double superphosphate is essentially the same as in the manufacture of ordinary superphosphate, except that finely ground phosphate rock is mixed with phosphoric acid instead of with sulfuric acid. Some of the details of these processes will be given in this report in the sections on history.

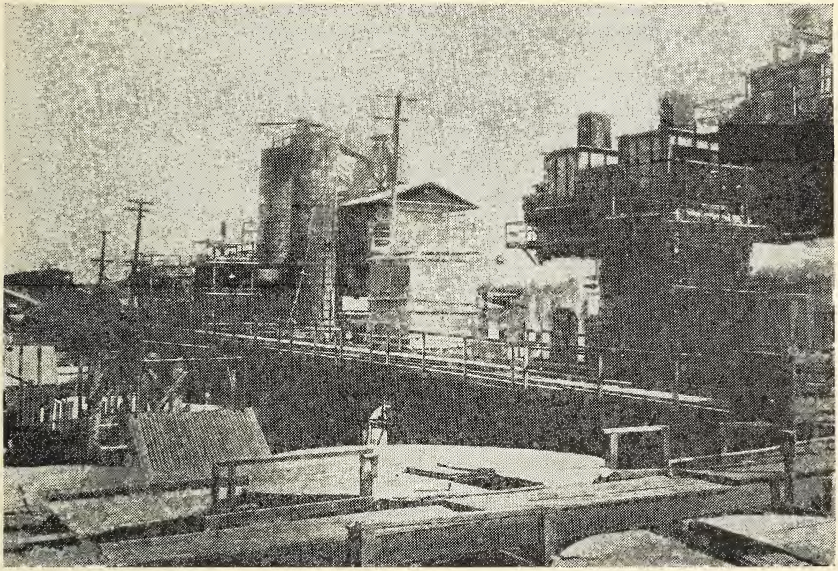

Figure 1.—Electrical precipitators formerly used in connection with an electric furnace process to make phosphoric acid. (Federal Phosphorus Co., Anniston, Ala.)

Marshall, Rader, and Jacob (*21*)[2] studied the effects of temperature, size of particle, strength of acid, time of reaction, proportion of acid to rock, and other factors with about a dozen different typical samples of phosphate rock. The best conversions obtained were with phosphoric acid containing 55 to 65 percent H_3PO_4 at 50° to 60° C. after 4 days' curing.

For additional information on the wet process see Voss (*40*), Kausch (*18*), Tsyrlin (*37*), and Voskresenskii, Milovanova, and Remen (*39*). For details of furnace processes see Ross, Carothers, and Merz (*31*), Waggaman, Easterwood, and Turley (*42*), Ross, Mehring, and Jones (*32*), Jacob (*15*), Waggaman and Easterwood (*41*), Easterwood (*10*), Curtis (*5, 6*), Curtis and Miller (*7*), Curtis, Miller, and Newton (*8, 9*), and Royster and others (*33*). Elmore and Farr (*11*) and Bassett (*2*) give phase-rule studies on the system, calcium phosphates, water, and phosphoric acid. Schmalfuss and Werner (*34*) give some principles that may be of interest in the preparation of high-analysis phosphatic fertilizers.

PATENTS

Many patents have been issued on processes and modifications of processes for manufacturing double superphosphate. A summary of all such patents issued by the United States up to 1925 is given by Waggaman and Easterwood (*41*). Kausch (*18*) gives a list of patents relating to double superphosphate issued by all countries up to about 1927. The principal patents relating to the preparation of double superphosphate issued by the United States prior to 1943 are listed in table 1.

TABLE 1.—*List of United States patents relating to the manufacture of double superphosphate*

Patent No.	Year of issue	Patentee	Title of patent or object
389,566	1888	Glaser, Charles	Production of concentrated phosphate fertilizer.
417,820	1889	do	Production of double acid phosphate.
459,575	1891	do	Production of phosphoric acid of growing strength without evaporation.
496,687	1893	Hoffman, P. C	Rapid production of soluble phosphate.
540,124	1895	VanRuymbeke, J	Process for producing phosphoric acid.
629,996	1899	Holtmann, J	Apparatus for making phosphoric acid.
631,181	1899	Schuler, G	Production of double acid phosphates free from pyrophosphoric acid.
655,458	1900	Saxl, H	Production of concentrated phosphate.
1,047,864	1912	Washburn, F. S	Production of phosphoric acid with electric furnace.
1,078,887	1913	Willson, T. L., and Hoff, M. M.	Method of making double superphosphate.
1,083,429	1914	Brunswig, F	Production of phosphoric acid.
1,351,672	1920	Meigs, C. C	Production of double superphosphate.
1,475,959	1923	Myers, H. H	Process for the production of double superphosphate.
1,706,101	1929	Blumenberg, H., Jr	Process for production of nonhygroscopic phosphates and the resulting product.
1,780,620	1930	King, W. B	Double superphosphate manufacture.
1,790,502	1931	Hechenbleikner, I	Process of manufacturing monophosphates of alkali metals and alkaline earths.
1,851,210	1932	Palazzo, F. C., and Palazzo, F.	Process for producing mixtures of monocalcium and dicalcium phosphates.
1,869,879	1932	Balz, O., and Wagner, W.	Decomposition of crude phosphates.
1,889,949	1932	Clark, C. B	Concentration of weak phosphoric acid.
1,891,007	1932	Palazzo, F. C	Process for producing calcium phosphates rich in P_2O_5 soluble in water.
1,902,648	1933	Larsson, Markus	Method of leaching raw phosphate.
2,021,671	1935	Skinner, L. B	Manufacture of superphosphates.
2,053,266	1936	Curtis, H. A	Manufacture of calcium phosphates.
2,067,538	1937	MacIntire, W. H	Process for making double or treble superphosphate.
2,070,582	1937	Curtis, H. A	Making superphosphate.
2,086,565	1937	MacIntire, W. H	Making calcium phosphate fertilizer.
2,148,209	1939	Loiseau, H., and Cartigny, G.	Method for the manufacture of superphosphates.
Reissue, 20,994	1939	Larsson, Markus	Method of treating raw phosphate.

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[2] Italic numbers in parentheses refer to Literature Cited, p. 22.

The first patent in this country on a process for making double superphosphate was No. 389,566, issued to Charles Glaser in 1888. This and the next three patents listed in the table were assigned to the American Phosphate & Chemical Co., of Baltimore. W. B. King, in United States Patent No. 1,780,620, describes the process said to have been used in 1930 in the Armour double superphosphate plant.

HISTORY OF PRODUCTION ABROAD

Double superphosphate was first made commercially in Germany. The facts relating to its early history were for the most part learned from Voss (*40*) and Kausch (*18*). A few of the facts given here were gleaned from early agricultural experiment station publications, various trade journals, and correspondence.

The purpose of the first companies that made double superphosphate was to find a practical method of utilizing the Lahn phosphorite. This phosphate, which up to then had no commercial value, is entirely unsuited to the manufacture of ordinary superphosphate, owing to its low P_2O_5 and high iron-and-aluminum content. Experiments showed that dilute sulfuric acid up to around 16 percent H_2SO_4 converted the P_2O_5 of this rock into phosphoric acid (H_3PO_4) without dissolving harmful quantities of iron and aluminum.

In 1870 the firm of H. and E. Albert started a small-scale plant at Biebrich, Germany, to manufacture double superphosphate. It was not successful at first, because no satisfactory way was found to separate the phosphoric acid from the calcium sulfate (gypsum) which is left as an insoluble residue. After numerous experiments this company developed a filter press that served very well for the purpose. Thus the first actual commercial production began early in 1872, and the business was profitable from the start. Another company, Müller, Packard & Co., began production later in the same year at Wetzlar, Germany. Somewhat later the Ohlendorff Guano Works, also in Germany, began making double superphosphate.

Before 1884, the wet process was usually carried out as follows: A batch consisting of 1 or 2 tons of finely ground phosphate rock, concentrated sulfuric acid, and wash liquor from a previous batch was run into a pine vat and stirred continuously for several hours. The solution of concentrated H_2SO_4 and wash liquor had a density of 16° B., and its quantity was gaged so that it contained three equivalents of SO_3 to one of P_2O_5 in the rock. Owing to the heat of dilution and chemical reaction the liquid acquired a temperature of about 70° C. (158° F.). After about 3 hours the entire batch was pumped into filter presses and washed first with the last wash liquor from a previous batch and then with tap water. The filter-press residue, known as phosphatic gypsum and containing 2 to 3 percent P_2O_5 and 40 to 45 percent water, was dumped. The dilute phosphoric acid separated from the filter presses was a straw-yellow solution of about 12° B., containing 8 to 10 percent P_2O_5, 0.2 percent SO_3, 0.4 percent CaO, and 0.3 percent $Al_2O_3+Fe_2O_3$. After settling for a time it was pumped into a concentrator. At first lead steam coils were used but without success. Then bottom heat was employed with gas flames. As water was evaporated it was carried away with compressed air blown through the solution and more dilute acid added until the concentration after about a week was raised to between 50 and 60 percent H_3PO_4. At the Albert plant in Biebrich five concentrators, each holding about 9,500 gallons, were operated so that each reached a maximum concentration on a different day. This plant produced an average of about 40 tons of concentrated acid daily. The evaporation of the 200 tons of water consumed about 26 tons of coal daily. The strong acid was then used to make double superphosphate in a mixing pan in the same way that sulfuric acid was used to make superphosphate. The mixing required 5 to 10 minutes, after which the batch was dumped into a den to cure.

In 1884 it was learned that the percentages of iron and aluminum oxides, hydrofluoric acid, and other constituents dissolved by the dilute acid increased with temperature and time of digestion. It then became customary to dilute the sulfuric acid with wash water from a previous batch and cool it before treating the ground rock to make phosphoric acid.

Since European countries had no deposits of phosphate rock suitable for making normal superphosphate and suitable imported materials were expensive, the double superphosphate process continued to be a success until high-grade imported rock became very cheap. By 1900, 12 companies in Europe were engaged in making double superphosphate on a large scale, but the number began to dwindle at about this time so that by 1919 only 4 or 5 plants survived. The competition from 18- to 24-percent grades made from cheap high-grade rock was too great for a number of these plants. Two plants, however, the La Nouvelle Montagne, in Belgium, and the Vereenigde Chemische Fabrieken, at Utrecht, Netherlands, are known to have been exporting this material to the United States up until 1939, when exports were cut off by the war; some other plants may have continued operation, but their names or location have not been learned.

Lehrecke (*20*) in 1937 described several recent European developments in the manufacture of phosphoric acid, such as the Landskrona Belt-Filter, the Autoclave Process, and the Open Anhydrite Process, before the Hamburg meeting of the International Superphosphate Manufacturers' Association. He concluded his address with the following statement: "It is now possible to produce phosphoric acid of high concentration so cheaply, at least in larger plants, that double or triple superphosphate can be manufactured at such prices, per unit of P_2O_5, as to approach very nearly that of the unit price of P_2O_5 in superphosphate. Moreover, the slightly higher cost can practically be disregarded, in view of the lower costs of transportation and packing."

In 1936 a small plant was constructed at Kirovsk, in the Soviet Union, to make 50-percent superphosphate. This plant uses elemental phosphorus produced electrically.

Double superphosphate has been produced in Japan since 1930, or possibly earlier than that, but few of the details are available. The Sumitomo Kagaku Kogyo is believed to have been making it for several years at their Niihama plant in Ehime Prefecture. About 3,000 long tons of 40-percent material were exported from Japan to the United States in the 3 years 1935–37.

HISTORY OF AMERICAN PRODUCTION

The first company to manufacture double superphosphate in the United States was the American Phosphate & Chemical Co., of Baltimore, Md., founded by the Dampmann interests. Dr. P. C. Hoffman, who later supervised the erection of the Virginia-Carolina Chemical Corporation's double superphosphate plant at Charleston, S. C., served as chemist for this company.

The fertilizer control reports of a number of States for 1891 and 1892 give analyses of their product, and it was registered for retail sale in Maryland in 1891. It appears, therefore, that production began in 1890 or 1891.

The fact that no analyses were reported later than 1892 does not necessarily mean that production stopped in that year, because practically the entire output was sold to manufacturers for use in mixed fertilizers, and State inspectors collect only samples of fertilizers sold in the retail trade. It is thought by several of the older members of the industry in Baltimore that double superphosphate was made by the American Phosphate & Chemical Co. for a few years only and that the entire production was probably less than a thousand tons. Table 2 shows the analysis of the composition of superphosphate prepared from figures given for samples of this company's material in the analysis reports of the Maryland and Connecticut Fertilizer Control Laboratories for 1891 and 1892.

TABLE 2.—*Composition of superphosphate manufactured by the American Phosphate & Chemical Co.*

Item	Percent	Item	Percent
Water-soluble P_2O_5	35.69	Silica (SiO_2)	6.37
Citrate-soluble P_2O_5	5.36	Moisture	6.25
Insoluble P_2O_5	.80	Water of constitution and undetermined	22.31
Lime (CaO)	17.78		
Sulfur trioxide (SO_3)	5.44	Total	100.00
Chlorine (Cl)	(1)		

[1] Trace.

The American Phosphate & Chemical Co.'s product was guaranteed to contain 40 percent available P_2O_5, and the actual content of a number of samples analyzed in State laboratories varied from 40.66 to 41.38 percent.

The second plant to make double superphosphate in this country was that of the Goulding Fertilizer Co., at Pensacola, Fla.[3] This company was a subsidiary of W. & H. U. Goulding, Ltd., of Dublin, Ireland, which still is one of the largest manufacturers of fertilizers in the British Isles. The manager of the company was sent to Germany in 1897 to consult engineers and obtain specifications for the plant, which was built at Pensacola in 1898. The plans included mixing apparatus, pumps, filter presses, heating, and concentrating apparatus for the dilute phosphoric acid, and drying equipment for the final product. The specifications called for mixing 1,000 pounds of finely ground phosphate rock with 1,150 pounds of 54° B. sulfuric acid (H_2SO_4) diluted with water to 16° B. just before mixing with the rock. The mix was to be agitated for 30 to 40 minutes and then pumped through filter presses. The liquor was to be concentrated by direct heat to 52°. No specific directions were given for the second step of the process, namely, mixing phosphate rock with phosphoric acid.

When the Goulding plant began to operate, the product contained only about 35 percent available P_2O_5 and was too hygroscopic. It was therefore necessary to do considerable research work to discover how to make a satisfactory product. This was finally learned, and, as commercially operated, 1,000 pounds of high-grade Peace River pebble phosphate was treated with 3,900 pounds of 54° B. phosphoric acid and allowed to cure in a den for 24 to 48 hours. It was then passed through a drying tunnel, using coke as the source of heat.

In 1898 this concern shipped between 50 and 100 tons of double superphosphate to London. Shipments of a few hundred tons annually continued during the next 2 or 3 years. This product contained 45.22 percent water-soluble P_2O_5, 2.11 percent citrate-soluble P_2O_5, and 5.90 percent moisture. Little or none of it was sold in the United States. Production of double superphosphate at this plant ceased about 1901. The following statement by Monroe and Chatard *(26, p. 564)* appears in the report of the 1900 census:

"Examination of the reports shows that only a comparatively small quantity of 'concentrated phosphate' is made, although it would seem that there ought to be a considerable demand for this product which is so largely made in England, France and Germany."

This is followed by a discussion of the process then in use for making double superphosphate.

The Virginia-Carolina Chemical Corporation's plant,[4] at Charleston, S. C., was the first one built of those now in operation in this country. (See fig. 2.) Dr. P. C. Hoffman, formerly with the American Phosphate & Chemical Co., supervised the erection and operation of this plant, which had an initial annual capacity of 5,000 tons. Production began in March 1907, and about 2,500 tons of double superphosphate were made during the first year of operation. South Carolina rock testing 60 to 64 percent B. P. L.[5] was first used to make a final product containing 44 percent available P_2O_5. Later Florida hard rock was used to make a higher grade product. This plant used essentially the same process in the beginning that is now in use there and was successful from the start. The plant began with diaphragm pumps for pumping the acid, and the concentrating tanks were heated with producer gas. The first method of drying the superphosphate was in a tunnel heated by the draft from steam boilers. These features have been replaced by more efficient devices.

Although the electric furnace had been used for many years to make phosphorus and phosphoric acid for other than fertilizer purposes, its first use to make acid specifically for double superphosphate manufacture was by the Piedmont Electro-Chemical Co., at Mount Holly, N. C., in 1914.[6] The plant was built in 1912 in order to use secondary power that it was believed would be available from a plant of the Saginaw River Dam. The original electric furnace was a large unit with three 20-inch carbon electrodes. As the cheap power expected did not materialize, the plant was abandoned in 1916, after having produced between 10,000 and 12,000 tons of double superphosphate and from 2,000 to 3,000 tons of ammonium phosphate.

The first use of the blast furnace to prepare phosphoric acid for production of double superphosphate began in 1931 at the Nashville, Tenn., plant of the Victor Chemical Works. No phosphoric acid has been made by the blast-furnace process for use in superphosphate manufacture for several years.

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[3] The facts concerning this company were supplied only a short time before his death by W. DeC. Kessler of Montgomery, Ala., who was secretary, treasurer, and manager of the company during the period under review.

[4] The data about this plant were supplied by Edward Ryland, vice president of the corporation.

[5] Bone phosphate of lime; to convert B. P. L. to P_2O_5 multiply by 0.4458.

[6] Data supplied by A. M. Webb, who was employed on the project.

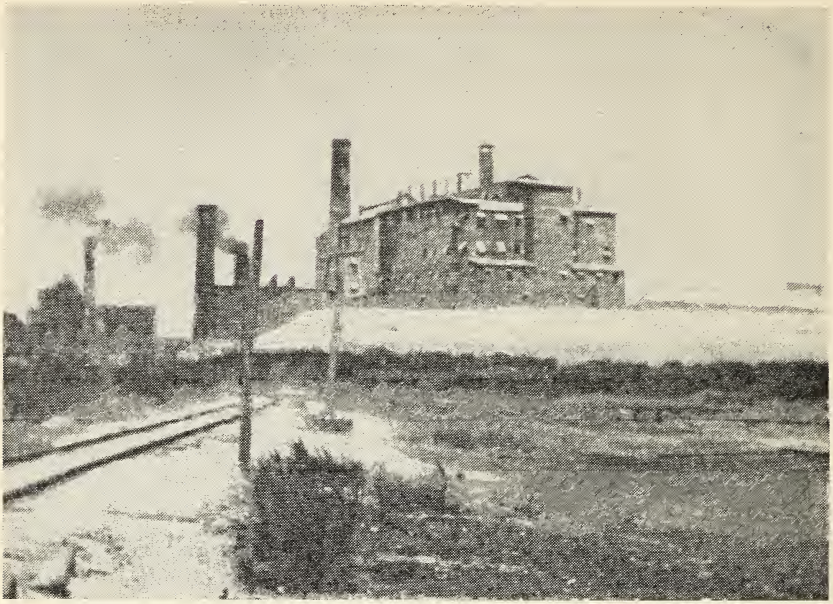

FIGURE 2.—Double-superphosphate plant of the Virginia-Carolina Chemical Corporation, Charleston, S. C., first one built of those now in operation in this country. The large light-colored mass in front of the building is the gypsum dump.

TABLE 3.—*North American double-superphosphate plants equipped to produce their own acid*

Name of owner	Location of plant	Phosphoric acid process used	Year production began
American Phosphate & Chemical Co	Baltimore, Md	Wet	[1] 1890
Anaconda Copper Co	Anaconda, Mont	do	1920
Armour Fertilizer Works	Columbia, Tenn	do	1929
Consolidated Mining & Smelting Co	Trail, British Columbia.	do	1931
Davison Chemical Corporation	Baltimore, Md	do	[2] 1922
Federal Phosphorus Co	Anniston, Ala	Electric	[3] 1924
Goulding Fertilizer Co	Pensacola, Fla	Wet	[4] 1898
International Minerals & Chemical Corporation.[5]	Wales, Tenn	do	1927
Mountain Copper Co	Martinez, Calif	do	[6] 1910
Phosphate Mining Co	Nichols, Fla	Electric and wet	1938
Piedmont Electro-Chemical Co	Mount Holly, N. C	do	[7] 1914
Tennessee Valley Authority	Wilson Dam, Ala	do	1934
United States Phosphoric Products Division, Tennessee Corporation.[8]	Tampa, Fla	Wet	1924
Victor Chemical Works	Nashville, Tenn	Blast furnace	[9] 1931
Do	Mount Pleasant, Tenn.	Electric	1937
Virginia-Carolina Chemical Corporation	Charleston, S. C	Wet	1907

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[1] Begun about 1890 and discontinued about 1893.
[2] Discontinued in 1925 and resumed with a new process in 1941.
[3] Discontinued in 1925. This operation was only a temporary means of utilizing surplus phosphoric acid.
[4] Discontinued about 1901.
[5] Originally the International Agricultural Corporation.
[6] Discontinued in 1912.
[7] Discontinued about 1916.
[8] Originally the United States Export Chemical Co. Plant purchased by the United States Phosphoric Products Corporation in 1927. Now a division of the Tennessee Corporation.
[9] Discontinued late in 1939 or early in 1940.

The Mountain Copper Co.[7] produced a relatively small tonnage of double superphosphate in 1910 at its plant at Martinez, Calif., with phosphoric acid produced by the wet process. None was produced in 1911, because the production in 1910 was not sold out until 1912, when more was produced. The operation was then abandoned because of difficulty in marketing the product at a profit.

Johnson and Cole *(17)* give details of the wet process and of plant equipment first used in an experimental plant by the Anaconda Copper Co. in August 1922 at Anaconda, Mont. This first plant was superseded a year or two later by a larger one. The details of the materials used and of the process, equipment, and product are given by Larison *(19)*.

The plant of the Consolidated Mining & Smelting Co., at Trail, British Columbia, began production of double superphosphate early in 1931 and was in regular operation about 6 months later. This plant has shifted operations so that it has produced at different times phosphoric acid, double superphosphate, or ammonium phosphate, whichever was in demand. The details and processes of this plant are described by Weber *(43, 44)*.

Table 3 gives a list of all known producers of double superphosphate in North America who made their own phosphoric acid, whether still in operation or not, as well as plant locations and dates of first operation. A few fertilizer companies, in addition to those listed, have made double superphosphate on a small scale with purchased phosphoric acid, both fresh and spent. One source of this spent acid is the synthetic resin industry.

PRODUCTION STATISTICS

The production of double superphosphate in the United States was ascertained by sending requests for figures to all known producers. Actual production figures were obtained for all plants except three that have long since ceased making this material. For two of these, estimates were obtained from former officials; no figures were obtainable for the American Phosphate & Chemical Co., but this concern probably made less than 1,000 tons and ceased operations before 1900. The total production in the United States for all years prior to 1907 was probably about 2,000 tons. The production figures by years beginning with 1907 are given in table 4. These figures include the

TABLE 4.—*United States production and shipments of double superphosphate, by years, 1907–43*

Year	Production [1]	Shipments [2]	Year	Production [1]	Shipments [2]
	Short tons	*Short tons*		*Short tons*	*Short tons*
1907	1,800	1,000	1926	37,082	36,604
1908	2,750	2,500	1927	36,856	36,589
1909	4,922	5,000	1928	68,313	60,587
1910	6,177	6,177	1929	80,266	73,107
1911	6,446	6,446	1930	95,198	89,224
1912	7,089	6,589	1931	48,287	43,725
1913	8,510	8,460	1932	24,192	24,524
1914	8,811	9,931	1933	29,542	31,046
1915	9,018	9,118	1934	69,552	51,618
1916	8,977	7,797	1935	91,059	81,781
1917	7,866	9,366	1936	129,746	111,810
1918	12,604	12,604	1937	166,144	170,572
1919	14,277	14,277	1938	186,920	179,520
1920	15,256	12,880	1939	272,750	258,451
1921	9,249	7,050	1940	329,046	343,539
1922	8,385	6,070	1941	317,990	342,529
1923	15,306	14,997	1942	314,430	318,090
1924	30,426	22,625	1943 [3]	294,144	301,363
1925	36,599	45,587			

[1] Actual tonnage produced, except for 1943.
[2] Includes material consumed in manufacturing operations at point of production.
[3] The tonnages for this period are on a basis of 45 percent available P_2O_5.

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[7] Data supplied by L. T. Kett.

production by the Tennessee Valley Authority and a small production from purchased acid by several fertilizer manufacturers.

The present capacity of the industry is estimated at about 450,000 tons annually, and this will be increased with the completion in 1945 of a plant at Houston, Tex., with a capacity of 70,000 tons. Stocks in the hands of producers on June 30, 1943, were reported as 50,344 tons (basis 45 percent). This checks closely with the 51,652-ton difference between the 2,670,585 tons produced and the 2,618,933 tons shipped or consumed in manufacturing, as reported in this study for the entire period covered by the history of the industry in this country up to that time.

About two-thirds of the acid consumed in making double superphosphate in 1942 was produced by the wet process and one-third by the electric furnace process. The average grade of the material produced was 43.5 available P_2O_5 in 1927, 44.7 in 1932, 45.6 in 1937, and 46.5 percent in 1942. The average grade made with furnace acid in 1942 was 48 percent and of that prepared with wet-process acid 45.7 percent.

IMPORTS

Although published import statistics of the United States do not show figures for concentrated superphosphate separately until 1935, something about earlier imports was learned from the export statistics of Germany, from literature on fertilizers, and from correspondence with the firm of H. J. Baker & Bro., of New York. The earliest imports into the United States were probably in 1877. An average of a few hundred tons annually was imported from Europe from 1883 to 1899. These shipments came chiefly from Ipswich, England, and from Germany. One of the principal brands during these years was known as Packard's double superphosphate, which seems to have disappeared from the United States market about 1901. Nothing definite could be learned about imports between 1901 and 1925, although it is known that some came into the country during these years. The unpublished records of the United States Bureau of Foreign and Domestic Commerce show that about 300 tons of concentrated superphosphate were imported in 1925, chiefly from Austria. It is definitely known that double superphosphate was imported from Canada each of the 11 years 1932–42. The imports of concentrated superphosphate, which include some material other than double superphosphate, for the years 1935 to 1942, inclusive, taken from the annual volumes of Foreign Commerce and Navigation of the United States, issued by the United States Bureau of the Census *(38)*, are shown in table 5 More than half of this tonnage came from the Netherlands and the rest from Canada, Belgium, and Japan.

TABLE 5.—*Imports of concentrated superphosphate, 1935 to 1942, inclusive*

Year	Amount	Value	Year	Amount	Value
	Short tons	*Dollars*		*Short tons*	*Dollars*
1935	3, 724	84, 022	1939	5, 537	165, 303
1936	2, 512	61, 224	1940	3, 208	39, 982
1937	14, 733	251, 051	1941	5, 973	133, 835
1938	9, 415	181, 224	1942	11, 977	300, 342

EXPORTS

The exports of double superphosphate are not known exactly, because the published figures include all superphosphates. It is known, however, that small lots have been shipped to various countries all over the world.

Ordinary superphosphate in the past 10 years has had an export value of $8 to $20 a ton, while double superphosphate has been valued at $30 to $75 a ton, according as the shipments were in retail or wholesale trade. Thus, shipments recorded in Foreign Commerce and Navigation of the United States (*38*) can be easily identified if they consist wholly of one or the other type of superphosphate. The exports listed in table 6 were estimated by comparing the value with the tonnage of each shipment, as given in the annual volumes of that publication, both by countries and by customs districts. These exports, however, are minimum values, because small shipments of double superphosphate recorded with large quantities of ordinary superphosphate would not be revealed in this way.

TABLE 6.—*Estimated disposition of double superphosphate*[1] *by uses*

Year	Direct use in agriculture			In manufacturing	Exports	Total
	Commercial distribution	Tennessee Valley Authority demonstrations	Agricultural Adjustment Agency grants			
	Short tons	*Short tons*	*Short tons*	*Short tons*	*Short tons*	*Short tons*
1925	15,000	----------	----------	25,300	3,700	44,000
1926	13,000	----------	----------	22,800	1,200	37,000
1927	8,000	----------	----------	27,000	2,000	37,000
1928	14,000	----------	----------	42,000	4,000	60,000
1929	17,700	----------	----------	47,300	8,000	73,000
1930	17,000	----------	----------	60,000	12,000	89,000
1931	15,300	----------	----------	28,000	700	44,000
1932	11,600	----------	----------	13,000	400	25,000
1933	8,800	----------	----------	23,000	200	32,000
1934	15,000	----------	----------	37,000	1,000	53,000
1935	14,500	14,000	----------	56,000	500	85,000
1936	16,700	27,000	----------	68,700	1,600	114,000
1937	31,000	18,000	25,000	92,000	2,000	168,000
1938	31,300	13,000	75,000	81,400	1,300	202,000
1939	40,000	18,000	124,000	85,400	2,600	270,000
1940	48,500	18,000	177,000	95,100	5,400	344,000
1941	51,800	20,000	95,000	128,200	53,000	348,000
1942[2]	55,500	21,000	9,600	143,900	100,000	330,000
1943[2]	48,000	21,000	0	86,000	150,000	305,000

[1] Includes imported material but not concentrated superphosphate grades under 38 percent available P_2O_5.
[2] Preliminary; subject to revision.

USES

The principal uses of double superphosphate are: (1) To increase the grade of run-of-pile ordinary superphosphate; (2) to make high-analysis grades of mixed fertilizers; (3) to serve in certain regions as an economical source of available P_2O_5 directly in agriculture; and (4) to adjust the grade of low-analysis mixtures when the P_2O_5 of goods already mixed is below a desired guarantee.

Run-of-pile ordinary superphosphate usually contains between 18.0 and 20.5 percent available P_2O_5. This material is used as it is for mixing operations in most plants. For sale to farmers and to some of the dry mixers, it has been graded to 14, 16, 18, 20, or 24 percent. In order to prepare a ton of 16-percent superphosphate, for example, from 19-percent run-of-pile material, 316 pounds of filler (limestone, sand, or other ingredients) must be added to 1,684 pounds of the higher grade goods. The 316 pounds of filler not only costs money but its addition to the superphosphate adds about one-sixth to the cost to the consumer of bagging, transportation, and handling. On the other hand, in order to raise 19-percent superphosphate to 20-percent grade, for example, 77 pounds of 45-percent superphosphate can be mixed with 1,923 pounds of the lower grade.

A unit of P_2O_5 in the form of double superphosphate usually costs the fertilizer manufacturer more than a unit in run-of-pile ordinary superphosphate, but less than the retail price of a unit of P_2O_5 in ordinary superphosphate. Double superphosphate is the ideal material to adjust upward the grade of any ordinary superphosphate. These two materials mix readily and blend perfectly because they have almost identical physical properties. The final product, usually 20-percent superphosphate, not only bears the total cost easily but also is practically everywhere cheaper per unit of P_2O_5 to the consumer than lower grades. Manufacturers have used double superphosphate to raise the grade of run-of-pile material ever since 1877. Its use for this purpose has been rapidly growing in the past 10 years and will probably continue to increase.

The second use of double superphosphate is as a source of P_2O_5 in the manufacture of high-analysis mixtures. This use began in recent years and has grown rapidly. Even though in some cases it costs more to make such mixtures, the farmer at some distance from the point of manufacture can well afford to buy them, because the saving in bags, labor, and transportation reduces the cost per unit of plant food, even though the manufacturer receives a higher price per ton.

Manufacturers have used very small quantities of double superphosphate since 1882 to adjust the grade of ordinary mixtures where the P_2O_5 upon analysis is found to be slightly below the desired guarantee or to fill orders for small tonnages of grades not in stock by changing the analysis of grades or bases already on hand. This relatively small use in manufacturing is not likely to increase greatly, because of the growing tendency to standardize grades.

As an economical source of phosphorus for direct use in agriculture, the chief competitor of double superphosphate is 20-percent superphosphate, because in many of the zones established for fertilizer-pricing purposes the unit cost of P_2O_5 is lowest to the ultimate consumer in the 20-percent grade. In most zones south of New York and east of the Appalachian Mountains, which is the most heavily fertilized region in the United States, as well as in some other areas, double superphosphate has no advantage over 20-percent goods made at the same location. In certain other regions in which plants are advantageously located with reference to raw materials and freight costs, the double superphosphate has decided advantages in price.

Average freight costs per unit of P_2O_5 in several grades of superphosphate are about as shown in table 7. Thus it costs as much to ship a unit of P_2O_5 in 18-percent superphosphate 10 miles as it does to ship the same quantity in the form of 45-percent goods 200 miles.

Total distribution costs (bags, freight, labor, and other expenses) on 18-, 20-, and 45-percent superphosphate in recent years have averaged about 68, 60, and 27 cents per unit of P_2O_5, respectively. This means that even where a unit of P_2O_5 in double superphosphate costs 30 cents more in the factory pile, it can usually be sold at a lower price per unit at points more than 200 miles away than normal grades manufactured at the same location.

Table 7.—*Estimated freight costs per unit of P_2O_5 in several grades of superphosphate, at various distances from the point of manufacture*

Distance from plant (miles)	18-percent grade	20-percent grade	45-percent grade	Distance from plant (miles)	18-percent grade	20-percent grade	45-percent grade
	Cents	*Cents*	*Cents*		*Cents*	*Cents*	*Cents*
10	6	5	2	350	23	20	8
75	12	10	4	500	28	25	10
200	17	15	6	1,000	45	40	18

CONSUMPTION

Various statements in the fertilizer literature before 1921 indicate that nearly the entire supply of double superphosphate was consumed in manufacturing other fertilizers prior to that year. This use has continued to grow rather rapidly ever since, except for the depression years 1931–35, as may be seen in table 6. The consumption of commercial fertilizers increased 57 percent from 1934 to 1942, but that of double superphosphate used in making them increased 314 percent.

Double superphosphate has been used directly in agriculture by farmers for the same crops and purposes as ordinary superphosphate, chiefly in sections where long freight hauls from factories have made it more economical. The earliest indication found in this study of its use by farmers is a statement in the 1883 report of the Connecticut Agricultural Experiment Station (*3, p. 30*), as follows: "[Sample] 987 contains more than double the amount of soluble phosphoric acid [36.37 percent A. P. A.] usually found in high grade superphosphates and at $70 per ton retail we believe is the cheapest source of soluble phosphoric acid in our market."

Very little double superphosphate, however, was purchased as such by farmers until 1921. The Anaconda Copper Co. started an intensive sales campaign among farmers in the North Central States in 1920, and in 1921 farmers really began to buy this material in substantial quantities. At present relatively large quantities are used directly in agriculture in the Rocky Mountain States, as indicated in table 8. In general, large quantities of double superphosphate are used in States that consume very little other fertilizer and vice versa. Figures 3 and 4 show the distribution of total consumption between manufacturing and direct use in agriculture.

From 1937 to 1942 the Agricultural Adjustment Agency (formerly the Agricultural Adjustment Administration) furnished double superphosphate to farmers for use in a soil-building program. The total quantities disposed of by calendar years are shown in table 6, and the quantities by regions and by program years in table 9. Each program year begins in the fall of the previous calendar year. All of this material distributed in the calendar year 1937 was prepared by the Tennessee Valley Authority and contained an average of about

43 percent available P_2O_5. In later years increasing proportions of the material were obtained from the fertilizer industry, and the P_2O_5 content increased to 46.5 percent in 1938 and to about 47 percent in the following years. In 1942 the demands of Lend-Lease for

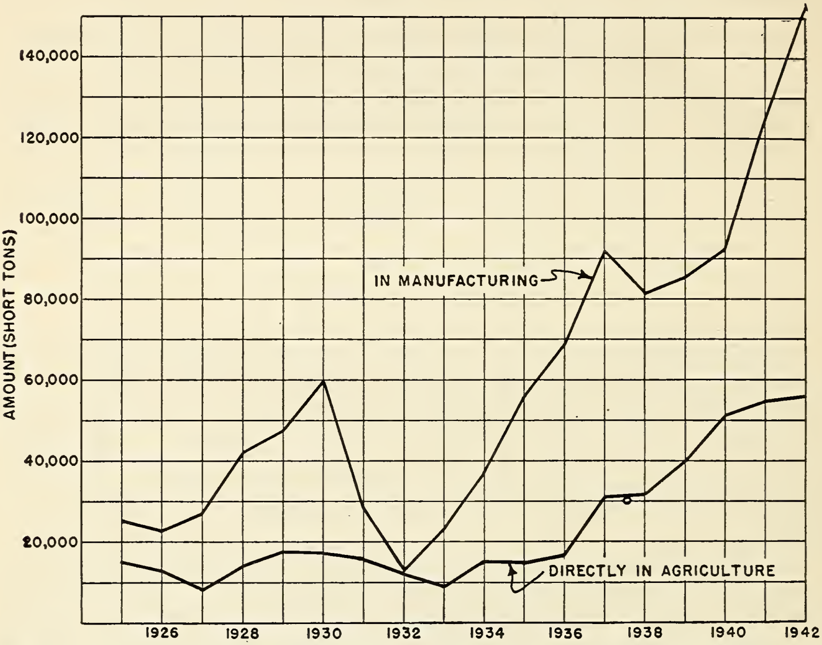

FIGURE 3.—Tonnage of commercially distributed double superphosphate consumed in manufacturing other fertilizers and directly in agriculture, 1925–42.

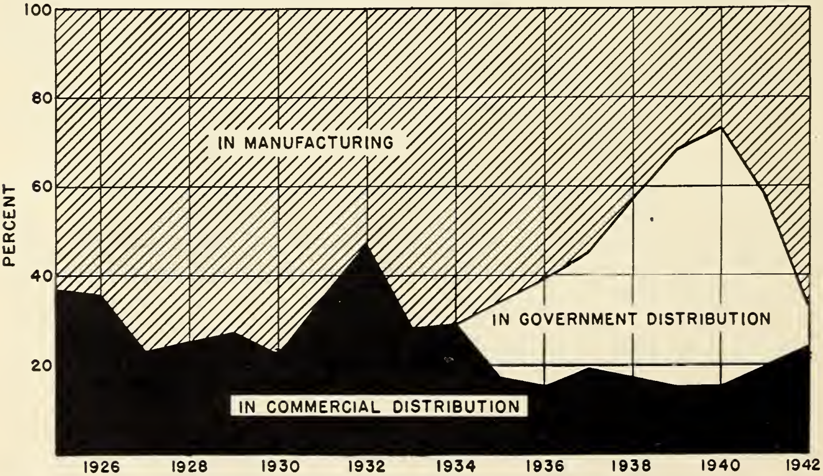

FIGURE 4.—Proportions of the total consumption of double superphosphate in manufacturing and directly in agriculture (Government and commercial distribution).

double superphosphate became so great that the Agricultural Adjustment Agency has since then confined its purchases to ordinary superphosphate.

Farm demonstrations of the value of double superphosphate have been made for test purposes on more than 25,000 farms since 1935 by the Tennessee Valley Authority. The total quantities thus distributed are shown by years in table 6, the bulk of this material having been consumed in Virginia, North Carolina, Georgia, Alabama, Mississippi, Tennessee, and Kentucky.

TABLE 8.—*Tonnage of double superphosphate sold to farmers through commercial channels, 1936–43, by States*

State	1936	1937	1938	1939	1940	1941	1942	Year ended June 30, 1943
Alabama	--------	--------	--------	--------	10	10	--------	--------
Arizona	80	668	223	147	1,044	3,325	3,000	660
Arkansas	--------	1	5	1	20	20	4	4
California	1,190	1,506	2,500	3,000	4,000	9,000	8,300	7,663
Colorado	1,364	2,621	1,340	2,500	2,100	2,800	4,000	4,613
Connecticut	--------	--------	25	32	50	50	172	73
Delaware	--------	--------	5	20	20	20	16	5
Florida	75	100	136	276	151	357	183	126
Georgia	--------	--------	2	6	15	10	0	0
Idaho	3,190	5,142	3,858	5,347	6,000	6,233	7,500	7,070
Illinois	112	425	4	11	10	88	112	290
Indiana	522	601	581	562	543	410	199	47
Iowa	34	50	61	45	80	313	208	323
Kansas	178	4,000	4,740	3,408	5,940	5,420	5,237	5,761
Kentucky	58	500	1,000	4,624	223	386	60	117
Louisiana	--------	5	1	--------	3	1	0	4
Maine	20	25	50	19	300	300	500	492
Maryland	20	24	25	38	6	5	4	5
Massachusetts	48	73	84	87	87	44	67	51
Michigan	255	391	316	421	154	235	100	79
Minnesota	586	1,726	1,001	1,438	3,402	3,833	2,638	3,831
Mississippi	--------	--------	--------	--------	--------	--------	--------	0
Missouri	216	457	591	843	1,190	1,119	1,177	1,279
Montana	3,249	3,912	3,654	4,128	5,000	3,459	4,000	4,471
Nebraska	593	1,430	1,200	1,222	1,500	1,500	1,500	1,411
Nevada	40	50	50	40	200	180	150	125
New Hampshire	5	5	10	8	12	23	24	24
New Jersey	50	177	30	4	48	50	75	3
New Mexico	465	711	732	829	1,364	1,497	2,326	2,085
New York	--------	--------	--------	--------	1	98	31	0
North Carolina	15	20	39	50	100	36	1,075	0
North Dakota	246	290	500	552	1,500	1,669	1,600	1,639
Ohio	179	811	518	570	1,759	332	181	130
Oklahoma	1	--------	1	3	1	12	20	6
Oregon	855	1,000	1,500	1,985	2,500	2,000	1,845	1,559
Pennsylvania	100	150	255	200	200	172	306	250
Rhode Island	5	5	10	19	20	18	10	0
South Carolina	10	20	50	136	7	36	2	1
South Dakota	119	107	200	255	250	200	300	350
Tennessee	--------	--------	74	302	350	400	300	29
Texas	171	220	150	1,163	846	647	1,259	845
Utah	699	1,870	1,242	1,835	2,000	1,412	2,500	2,642
Vermont	35	35	35	37	50	50	15	12
Virginia	--------	--------	1,179	147	150	150	92	0
Washington	64	40	1,000	1,469	2,500	2,000	2,246	2,045
West Virginia	--------	--------	50	55	100	205	120	0
Wisconsin	177	164	165	154	150	150	7	1
Wyoming	1,618	1,676	2,058	2,033	2,500	1,534	2,000	1,453
United States	16,644	31,008	31,250	40,021	48,456	51,809	55,461	51,574

TABLE 9.—*Tonnage and cost of double superphosphate distributed to farmers by the Agricultural Adjustment Agency by program years and regions*

Program year	Region	Quantity	Cost[1]
		Short tons	*1,000 dollars*
1938	Northeast[2]	7,962	304
	North Central[3]	-----	-----
	East Central[4]	56,771	1,847
	Southern[5]	2,214	69
	Western[6]	-----	-----
	Total	66,947	2,220
1939	Northeast[2]	15,872	698
	North Central[3]	617	23
	East Central[4]	92,237	3,358
	Southern[5]	10,620	379
	Western[6]	4,119	146
	Total	123,465	4,604
1940	Northeast[2]	20,805	790
	North Central[3]	15,219	579
	East Central[4]	112,158	4,038
	Southern[5]	18,617	668
	Western[6]	10,201	407
	Total	177,000	6,482
1941	Northeast[2]	185	7
	North Central[3]	22,542	902
	East Central[4]	60,352	2,446
	Southern[5]	22,632	865
	Western[6]	6,564	289
	Total	112,275	4,509
1942	Northeast[2]	-----	-----
	North Central[3]	-----	-----
	East Central[4]	12,723	441
	Southern[5]	-----	-----
	Western[6]	13,318	417
	Total	26,041	858

[1] Includes freight to destination.
[2] New England, New York, New Jersey, and Pennsylvania.
[3] Illinois, Indiana, Iowa, Michigan, Minnesota, Missouri, Nebraska, Ohio, South Dakota, and Wisconsin.
[4] Delaware, Maryland, Virginia, West Virginia, North Carolina, Kentucky, and Tennessee.
[5] South Carolina to Texas and Oklahoma, inclusive.
[6] All other States.

AGRICULTURAL VALUE

In the Atlantic Coast States double superphosphate is used principally on hay crops and pastures. In the Central States it is used chiefly on wheat and oats, and on clover, alfalfa, and other hay crops. West of the Mississippi it is used largely on sugar beets and alfalfa. It has been used also with success on the west coast on berries and other crops.

In comparison with other phosphatic fertilizers very few tests of double superphosphate were made with crops until comparatively recent years. Since 1934 large numbers of such tests have been made in many States, principally as a result of the activities of the Tennessee Valley Authority. Results of tests made up to about 1939 are summarized in a report by Ball (*1, pp. 29–41*). These tests were made by the agricultural experiment station staffs of 7 Southeastern States on several hundred soil types and with the following crops: Corn, cotton, cowpeas, soybeans, oats, wheat, potatoes, hay, and pasture. The Kentucky station reported that "ordinary superphosphate and triple superphosphate were about equally effective" in tests on each of the principal soil groups of the State. In 285 tests on cotton made in Alabama, including several soil types in each of the 6 soil regions of the State, 43-percent superphosphate gave a relative yield of 97 as compared with 100 for 16- or 20-percent superphosphate. The North Carolina station, as a result of experiments on a variety of crops and soils, states (*1, p. 38*) that "triple superphosphate, on most soils, appears to be a satisfactory source of phosphoric acid as compared with standard sources." The Tennessee station reports that "triple superphosphate proved to be a first-class source of phosphoric acid for fertilizer use on corn, wheat, potato, soybean, and cowpea." The Virginia Agricultural Experiment Station found that the average yield (without lime) from triple superphosphate on 19 different soils and with a variety of crops and crop rotations has been within 3 percent of the yield from 16- or 20-percent superphosphate.

The average relative yields from various treatments in more than 3,000 experiments with a wide variety of crops and soils, summarized by Ball *(1, p. 39)*, is in principle as follows:

	Percent
No fertilizer	64. 5
N+K, but no P	79. 9
16- or-20 percent superphosphate + N and K	100. 0
Double superphosphate + N and K	98. 0
Double superphosphate + N, K, and S	100. 9

For experimental results showing that double superphosphate also gives excellent results on western soils, see McGeorge (*22*), Green (*12*), Toevs and Baker (*35*), and Hinkle (*14*). Experiments made by these investigators seem to demonstrate that where soils are deficient in sulfur, normal superphosphate gives slightly higher yields, but that in most cases there is no significant difference between the results obtained with ordinary and double superphosphate.

O'Brien (*28*), found that both normal and double superphosphate improve the quality of grasses in the same way. For example, the use of equivalent quantities of both these materials increased the nitrogen content of pasture grasses about 20 percent.

Both normal and concentrated superphosphate have been used to prevent loss of nitrogen in fermenting animal manures. Midgley and Weiser (*23*) found that, compared with double quantities of 20-percent goods, 40-percent superphosphate was less efficient in preventing loss from cow manure. The untreated manure at the end of the fermentation period contained 7.3 percent, that treated with 40-percent superphosphate 22.4 percent, and that treated with 20-percent superphosphate 30.8 percent of the original nitrogen.

CHEMICAL COMPOSITION

Double superphosphate is essentially impure monocalcium phosphate prepared in a particular way. Generally it contains between 40 and 48 percent of available P_2O_5 and 18 to 22 percent lime (CaO), whereas chemically pure monocalcium phosphate contains 56.34 percent available P_2O_5 and 22.23 percent CaO. The principal impurities are calcium sulfate, fluorides, iron and aluminum phosphates, silica, and moisture. It also usually contains traces of a large variety of other elements, as indicated in table 10. The acid-soluble impurities from both batches of phosphate rock consumed when wet-process phosphoric acid is used, as well as most of those in the sulfuric acid, are concentrated in the double superphosphate.

The average available P_2O_5 content of 41 analyses of samples reported in the literature between 1877 and 1919 is 43.83 ± 0.23 percent, and of 998 samples

TABLE 10.—*Typical analyses of double superphosphate made with variously prepared acids from different phosphate rocks*

Constituent	Wet process acid from—							Blast furnace acid from Tennessee brown rock [1]	Electric furnace acid from Tennessee brown rock	
	Florida land pebble [1]	Florida land pebble [2]	Idaho rock [1]	Idaho rock [2]	Montana rock [2]	South Carolina rock [3]	Tennessee brown rock [1]			
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent* [4]	*Percent* [2]
P_2O_5	46.21	49.83	47.33	48.00	45.0	41.87	43.90	48.37	50.1	49.80
SO_3	3.19	1.81	2.74	3.03	3.6	5.44	6.50	2.10	----	.15
F	2.04	2.18	1.81	1.90	----	----	2.13	1.15	1.88	1.75
CaO	18.62	18.02	19.64	21.75	19.5	17.78	17.17	18.48	20.8	21.40
Fe_2O_3	2.04	3.92	.83	.84	1.9	----	3.48	1.25	.7	1.70
Al_2O_3	1.40		1.73	1.90	2.5	----	2.62	.98	1.8	1.40
MgO	----	.76	----	.60	.05	.15	----	----	----	.10
K_2O	----	.22	----	.09	0	----	----	----	----	.17
Na_2O	----	----	----	.19	.10	.10	----	----	----	.15
MnO	----	.11	----	.06	.016	----	----	----	----	----
CuO	----	----	----	.015	.01	----	----	----	----	----
B_2O_3	.009	----	.010	$<.01$	----	----	.008	.016	----	----
Cr_2O_3	----	----	----	.12	.05	----	----	----	----	----
As_2O_3	----	----	----	.023	----	----	----	----	----	----
V_2O_5	----	----	----	.32	.32	----	----	----	----	----
BaO	----	----	----	$<.01$	----	----	----	----	----	----
SrO	----	----	----	$<.01$	----	----	----	----	----	----
PbO	----	----	----	.002	.006	----	----	----	----	----
SeO_3	$<.0001$	----	.006	----	----	----	----	.0003	----	----
N	----	.014	----	----	----	----	----	----	----	----
TiO_2	----	----	----	.05	.10	----	----	----	----	----
SiO_2	----	3.31	----	3.60	5.5	6.37	----	----	3.2	3.20
Moisture	----	----	----	2.60	6.2	5.07	----	----	1.0	----
Free H_2O	3.50	4.10	2.67	----	----	----	1.24	.65	----	2.20
Crystal H_2O	3.63	----	2.70	----	----	----	6.26	4.76	----	----
Undetermined [5]	19.36	15.73	20.54	14.90	15.15	23.22	16.70	21.74	20.52	17.98
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Available P_2O_5	45.62	49.68	44.96	45.35	44.1	41.13	43.20	47.89	49.1	47.8
Water-soluble P_2O_5	38.30	42.16	41.30	39.00	----	35.22	29.90	42.20	40.3	----
Insoluble P_2O_5	.49	.15	2.37	2.65	.9	.74	.70	.48	1.0	2.0
Free acid P_2O_5	1.44	5.52	1.95	2.03	----	----	.78	.52	.2	----

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[1] Hill and Hendricks (*13*).

[2] Manufacturers' analyses obtained by correspondence.

[3] Connecticut Agricultural Experimental Station Annual Report (*4*).

[4] Newton and Copson (*27*).

[5] By difference. This consists mostly of the water of constitution of the monocalcium phosphate.

since 1920 is 44.30±0.04 percent. The standard deviation from this latter mean is 1.7 percent. The minimum and maximum values were 38.58 and 52.80 percent, respectively. The mode occurs at 44.2 percent. Of the 998 samples, 63.56 percent contained between 43.0 and 46.0 percent available P_2O_5.

The average available P_2O_5 content of superphosphate produced by manufacturers for the years 1927 to 1943, inclusive, was obtained. The resulting figures were weighted by the tonnage produced, to secure the percentage P_2O_5 in the product. The percentages for the different years are given in table 11.

TABLE 11.—*Percentage P_2O_5 content of superphosphates produced by manufacturers, 1927 to 1942, inclusive*

Year	Percent P_2O_5	Year	Percent P_2O_5
1927	43.84	1935	45.17
1928	44.05	1936	44.64
1929	44.38	1937	45.36
1930	44.47	1938	46.40
1931	44.85	1939	46.85
1932	44.87	1940	46.90
1933	45.19	1941	47.11
1934	45.43	1942	46.74

There was no significant change in the available P_2O_5 content of the samples reported in the literature from 1877 to 1927, but since then the P_2O_5 has gradually increased to a present average of about 47.0 percent.

The average insoluble P_2O_5 of 35 samples reported between 1877 and 1919 is 1.59±0.16; of 848 samples since 1920, 1.84±0.04. The mean of the probable errors, 0.16 and 0.04, is 0.10, and since the difference between 1.59 and 1.84 is less than 3 times the mean probable error, the difference shown between the two average insoluble P_2O_5 figures may be due to chance in the way the samples were selected.

The moisture content as reported in the literature has decreased from a mean of 7.84±0.22 for the earlier samples to 4.36±0.18 for the more recent ones. This indicates a decided improvement, which is believed to be due partly to the fact that recently manufactured material contains less free acid and is therefore less hygroscopic, and partly to better drying. The above moisture determinations for the most part were undoubtedly made on samples approaching equilibrium with the average of normal atmospheric conditions, but the atmospheric conditions vary from one place and from one season to another. Mehring and Cumings (*25*) found that a typical double superphosphate has the moisture content shown in table 12 when in equilibrium with the temperatures and relative humidities shown.

TABLE 12.—*Percentage of free moisture in double superphosphate when in equilibrium with various temperatures and relative humidities*

Temperature (° F.)	Relative humidity (percent)					
	40	50	60	70	80	90
50	--------	--------	4.34	--------	--------	--------
68	2.44	3.24	4.45	6.86	9.83	16.92
86	--------	--------	4.65	--------	--------	--------

In addition to the typical analyses shown in table 10, many more are available in the literature and in correspondence on file in the United States Department of Agriculture. These determinations were used to obtain the composite figures given in table 13. A study of all these determinations leads to the following conclusions: No consistent differences in composition between double superphosphates made with wet-process and furnace-process acid are found. On the average, furnace-process acid produces superphosphates containing less sulfates, but some samples thus made contain as high as 3 percent SO_3. The sulfur is derived in these cases both from the original phosphate rock and from the coke or coal used to reduce it. The fluorine content of wet-process material averages considerably higher than furnace-acid material; nevertheless some samples of the former contain less than certain samples of the latter. For the same type of rock, wet-process material usually is much higher in iron and aluminum content than furnace-process material.

TABLE 13.—*Composition of double superphosphates analyzed*

Constituent	Percentage			Number of samples
	Minimum	Maximum	Average	
P_2O_5	42.40	55.65	46.29	993
N	.06	.40	.26	5
B_2O_3	(1)	.084	.036	4
SO_3	.15	6.29	3.40	20
SeO_3	.00012	.00064	.00034	3
F	.20	3.74	1.56	77
Cl	0	(1)	(1)	3
Br	0	0	0	2
I	0	0	0	2
K_2O	0	.57	.35	9
Na_2O	.13	1.79	.97	9
CaO	16.20	26.40	19.99	102
MgO	.05	1.00	.38	10
BaO	0	0	0	4
SrO	0	(1)	(1)	4
Fe_2O_3	.70	3.48	1.36	27
Al_2O_3	.01	3.60	1.93	28
MnO	0	.060	.019	5
ZnO	(1)	.017	.005	4
CuO	0	.027	.015	11
PbO	0	.007	.0028	3
Cr_2O_3	0	.13	.075	10
As_2O_3	0	.150	.094	8
V_2O_3	0	.57	.37	9
TiO_2	0	.10	.05	3
SiO_2 and acid insoluble	.60	7.37	4.42	31
Free H_2O	.20	9.50	3.30	95
H_2O of crystallization	1.29	6.26	3.47	7
H_2O of constitution and undetermined			11.64286	(2)
Total			100.00	
Moisture	2.04	11.37	5.12	81
Water-soluble P_2O_5	18.85	50.63	39.41	168
Citrate-soluble P_2O_5	3.00	21.70	6.14	152
Available P_2O_5	38.58	52.80	44.47	1,039
Citrate-insoluble P_2O_5	0	8.45	1.77	993
Free H_3PO_4	.28	9.50	2.79	97

[1] Trace. [2] By difference.

Jacob and associates (*16*) analyzed 21 typical samples of double superphosphate prepared with wet-process acid by the principal American plants and found 1.65 to 3.90 percent fluorine, with an average of 2.44 percent. Similarly, 9 samples prepared from furnace acid were found to contain 1.08 to 1.75 percent fluorine, with an average of 1.43 percent. The B_2O_3 content of 4 samples of double superphosphate given in table 6 is 0.036 percent, or considerably higher than the average of 0.0106 percent found by Rader and Hill (*30*) on 4 other samples. These authors point out the possibility that high results may be obtained by storing samples in glass containers. Tremearne and Jacob (*36*) determined arsenic in 11 typical samples of double superphosphate and found 0.0011 to 0.0405 percent, with an average of 0.0074 percent. Rader and Hill (*29*) found the following quantities of selenium in representative samples of double superphosphate made from the kinds of rock indicated:

	Parts per million
Florida land pebble	<0.8
Tennessee brown	1.5
Idaho	4.0

The calcium, phosphorus, iron, aluminum, and moisture contents of materials made from eastern and western rocks average about the same. The proportions of all the constituents of double superphosphate except P_2O_5, CaO, and SO_3 are of about the same general order as those of the same constituents of 16- or 20-percent superphosphate.

PHYSICAL PROPERTIES

Double superphosphate is a gray, coarsely powdered material resembling ordinary superphosphate. Its physical properties that have a bearing on its use as a fertilizer are apparent density, or specific gravity, particle-size distribution, and drillability.

The apparent density was determined on 45 samples. The results on 8 samples of double superphosphate varied from 0.83 to 1.05, with an average of 0.96 ± 0.01; and on 37 of superphosphate from 0.71 to 1.05, with an average of 0.91 ± 0.003. The small probable errors of the averages show that most of the samples had apparent specific gravities close to the average. (The apparent specific gravity of fertilizer materials in general varies from about 0.3 to 2.0.) Thus it is apparent that for bag size, distributor-hopper capacity, and other practical purposes, all grades of superphosphate have about the same value.

Screen analyses were made on 10 typical samples of commercial 16- to 20-percent superphosphate and 7 samples of 40- to 46-percent goods. With one exception, each sample was obtained from a different manufacturer. The results are compared in table 14.

Table 14.—*Percentages of normal and double superphosphates of various screen sizes*

NORMAL SUPERPHOSPHATES

Grade percent	Description or source	Screen sizes						
		On 5	5–10	10–20	20–40	40–80	80–200	Through 200
16	Charleston, S. C	5.8	9.3	11.0	18.5	25.5	18.7	11.2
16	Atlanta, Ga	3.4	6.8	8.7	21.9	23.6	20.0	15.6
16	Charleston, S. C	1.8	6.6	8.4	23.2	24.9	20.0	15.1
16	Savannah, Ga	1.9	4.2	7.3	20.0	26.2	21.3	19.0
18	Norfolk, Va	5.6	8.0	8.9	12.3	19.4	27.4	18.4
18	Made about 1926 by unknown manufacturer	0	0	.1	24.2	14.6	12.3	48.8
20	Baltimore, Md	1.6	3.7	3.6	16.3	23.8	35.2	15.7
20	Columbus, Ohio	.1	5.2	20.1	26.3	24.1	12.1	12.1
20	Carteret, N. J	4.6	13.0	9.7	18.4	23.8	16.1	14.3
20	Granulated "Oberphos"	0	4.2	24.0	32.3	20.0	15.7	3.8
	Average	2.48	6.10	10.18	21.34	22.59	19.88	17.40
	DOUBLE SUPERPHOSPHATES							
43	Montana (made in 1926)	1.9	81.8	8.6	2.4	2.4	2.8	0.1
43	Tennessee (wet-process acid)	.4	29.9	22.1	22.8	14.3	7.4	3.1
44	Montana (made in 1935)	0	5.7	32.7	23.3	18.0	11.1	9.2
45	Baltimore, Md	.4	19.5	35.9	32.3	11.0	.6	.3
45	Florida (wet-process acid)	2.0	19.2	21.0	21.6	20.4	10.6	5.2
48	Tennessee (furnace acid)	0	5.5	18.1	5.4	23.6	22.3	15.1
48	South Carolina	.1	2.2	12.7	21.3	37.5	19.9	6.3
	Average	.69	23.40	21.59	19.84	18.17	10.67	5.61

In general, the double superphosphates are composed of coarser particles than the samples of ordinary grade tested, except the one specially granulated, owing no doubt to the fact that artificial drying is usually employed in making double superphosphate. This is generally carried out by passing the material through a revolving cylinder counter to a stream of hot gas. Such manipulation naturally granulates it more or less and thus produces a material of better drillability.

The drillability of fertilizers may be measured by their angles of repose when the apparent density remains fairly constant (*24*). Superphosphate and double superphosphate have approximately the same apparent specific gravity. Their drillability, therefore, may be compared by means of angle-of-repose measurements. Accordingly, the angle of repose was measured on typical samples of commercial superphosphate.[8] The angle of repose of 8 samples of double superphosphate varied from 35.4° to 44.2°, with an average of 38.6° ±1.0°. Similar results for 55 samples of normal superphosphate varied from 39.0° to 50.5°, with an average of 43.3° ±0.3°. In view of these results there can be little doubt that double superphosphate on the average has better drillability than 16- to 20-percent superphosphate that has not been granulated.

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[8] Most of these samples were made available by control officials of New York, New Jersey, South Carolina, Georgia, and Tennessee, to whom grateful acknowledgment is hereby made. The rest were materials purchased on the market by the Division of Soil and Fertilizer Investigations.

The drillability of any fertilizer changes with its moisture content, and this depends largely upon its hygroscopicity. The average double superphosphate is one of the least hygroscopic of all fertilizer materials. In controlled experiments (*25*) it was shown to be completely drillable when at equilibrium with atmospheres of normal temperatures and with relative humidity from 20 to 90 percent.

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